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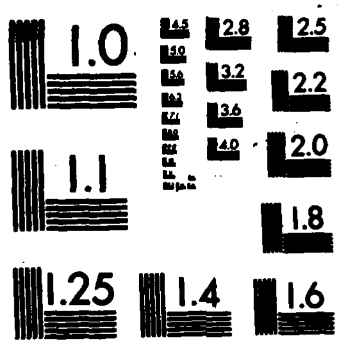

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Cyclic Deformation and Fatigue of Monocrystalline Ni-Base Superalloys

FINAL REPORT

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FINAL REPORT

"Cyclic Deformation and Fatigue of
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Grant No. DAAG-29-82-K0014

Summary

A report is provided of progress in studying cyclic deformation of precipitation-hardened alloy in a project supported by the army. This work concentrated on the stability of precipitates under cyclic deformation (Al-Ag, Al-Cu systems) and the strain-localization behavior of alloys containing shearable precipitates (Al-Cu, θ") and non-shearable precipitates (Al-Cu, θ'). Furthermore, the relationship of cyclic response in such simple systems to that of commercial alloy was also studied. The main results are ~~as follows:~~ reported. for the alloy systems and compositions chosen, precipitate dissolution did not occur. Rather fatigue-induced precipitation occurred in Al-Ag, even at 78K. Precipitate degradation did occur at room temperature in Al-Cu alloy containing θ" precipitates, causing strain localization and cyclic softening. The localized strains, ranging from 0.3 to 0.6, were found extremely high. Al-Cu alloy containing θ" precipitates, which are too large and widely spaced to be cut by the dislocations, was found not to show a plateau in its cyclic stress-strain curve, in contrast to the alloy containing θ" precipitates. The dislocation structures of the θ' alloy were dominated by arrays at the broad faces of the θ', and were markedly similar to those which form in pure metals ('maze' structure). Commercial alloys, including both Al and Ni based alloys, were found not to cause cyclic softening because of the

strain-homogenizing effect of their larger particles. Major progress was made in relating the cyclic deformation behavior of mono- and polycrystalline material.

Introduction

In the last ten years, major progress has been made in understanding the cyclic deformation of single phase metals. Thus the range of strain amplitudes in which persistent slip bands form was identified, the dislocation structures associated with these bands and other forms of cyclic deformation were explored and understood in large (if by no means complete) measure, and considerable advances have recently been made in understanding crack nucleation (Laird et al at Pennsylvania, Fine et al at Northwestern, Neumann et al at Dusseldorf and Mughrabi et al at Stuttgart).

During this same period, less attention was paid to the cyclic deformation behavior of precipitation hardened systems although notable advances were made by Fine et al, Haasen et al and by Gerold and coworkers at Stuttgart. About 5 years ago Laird proposed to the Army Research Office a program to exploit the techniques developed for single phase crystals for studying useful precipitation systems and commercial alloys based on such systems. The aim of the work has been to understand why the hardening precipitates are unstable to cyclic strain because this leads to cyclic softening in persistent slip bands (PSBs), strain localization and, therefore, fatigue failure at disappointingly low applied stresses. Both crack nucleation and propagation mechanisms are affected by such precipitate instability and PSB's.

The mechanisms of instability differ in different alloy systems and between commercial and experimental alloys. Thus Al-Zn-Mg alloys soften by precipitate dissolution(1), but Al-Cu alloys do not(2). In the latter system, Calabrese and Laird(2) suggested that the strengths of the shearable precipitates were reduced by disordering of their crystal structure by cyclic stressing. To check this idea, Al-Ag alloy containing GP zones, which are known to be disordered as formed and in the absence of cycling, was cycled to find out whether or not it underwent cyclic softening(3). It did not, and instead hardened by fatigue-induced precipitation to high flow stresses(3). In order to suppress such precipitation, we further proposed to strain-cycle Al-Ag alloy at 78K. Moreover, to study the localization of strain during fatigue, we proposed to cycle Al-Cu single crystals containing coherent θ'' precipitates and to measure the strain in individual PSBs by interferometry. We particularly wanted to study the changes in strain localization which occur during cyclic softening.

Another aim of this work was to relate fundamental studies on model precipitation hardened alloys to the behavior of commercial alloys. Accordingly, the cyclic response of polycrystalline model alloys and commercial alloys was proposed. The most obvious relation to results on model alloy single crystals was to monocrystalline nickel base superalloys and this also was proposed, and some studies were initiated. However, the work was found difficult, there were material supply difficulties, and the crucial Finney-type experiments were not possible. However, during our work on Al-Cu alloy, we obtained a breakthrough in interferometry technique

using replicas and this has opened up the possibility of studying nickel base alloys effectively. We therefore proposed last year to complete our work on model Al-Cu alloy and then to work exclusively on nickel-base superalloys. The studies of Al-Cu were successfully completed and good progress was made on superalloy.

Progress in the Total Grant

The study of Al-Ag alloy was done first(4). A low temperature strain cycling rig was built and Al-Ag alloy, suitably prepared with GP zones, was cycled at 78K. In support of the disordering hypothesis of cyclic softening, no cyclic softening was observed(4), and the experiment was thus considered valid in support of the disordering hypothesis. However, at the lowest strains studied, with consequent longest lives, fatigue-induced precipitation of γ'/γ precipitates was discovered by TEM(4). We interpreted this astonishing result on the consideration that vacancies would indeed be immobile at 78K but dislocation interactions would produce interstitial defects and these could cause the diffusion necessary to form the γ'/γ plates(4). Moreover, the γ'/γ plates formed close to pre-existing GP zones (and at their expense) so that the range of the diffusion required would be small.

It is interesting to recall that Polmear, in a series of studies, found significant improvements in mechanical properties, including fatigue, by small additions of Ag to Al-Zn-Mg alloy and more complex alloys based on this ternary. However, he never developed a sound explanation of the phenomena, so it would be interesting to explore this matter further. The investigator suspects that silver segregation and precipitation enhanced the

nucleation of the Zn/Mg particles but during this grant, we had no time or resources to study the problem.

Second, we studied extensively Al-Cu single crystals containing θ" shearable precipitates. For this study, we required very accurate control and recording of plastic strain. A new MTS machine was bought, modified and developed (a task which took three months and much bullying of the supplier to meet his own specifications). Even after this, recording strain down to 10^{-6} was inadequate and we therefore built our own system to bypass the MTS electronics. For accurate X-Y recording of the plastic strain at any part of the hysteresis loop, and especially at 0 stress where the anelastic strain is zero, we arranged to chop off the extremities of the loops and thus to limit the travel of the pen. This system has worked very well, and the results at low strains were obtained with it.

Just like polycrystals, Al-Cu single crystals harden to a peak and then cyclically soften. We were delighted to find that the cyclic stress-strain curve for Al-Cu, based on the peak hardening stress, shows a plateau rather like that for single crystals of pure copper. However the plateau extends from 10^{-5} to 10^{-3} (plastic strain amplitude), i.e. displaced a decade of strain lower than that of copper. This implies that the localized strains in precipitation hardened alloys are orders of magnitude greater than those in pure metals and in support of this we found that the lives of our Al-Cu single crystals are two orders of magnitude lower than those of copper single crystals tested at the same strains.

Believing that the lower limit of the plateau should correspond to a fatigue limit we stress-cycled specimens for large numbers of cycles at stresses lower than that of the plateau. We found that the slip remains fine and uniform in such specimens, that PSBs do not form, and that cyclic softening does not occur. However, the specimens were observed to fail eventually because we had left the extensometer attached so as to detect softening and the knife edges had sunk into the metal by cyclic creep. Cracks grew from these sites because the fatigue stress limit necessary to nucleate a crack is higher than that necessary to exceed the threshold stress intensity for growth of an "artificial" crack.

We have also compared the cyclic stress-strain curve of polycrystalline Al-Cu at low strains with that for monocrystalline Al-Cu, by dividing the stresses and strains of the polycrystalline alloy by Taylor's factor. We were pleased to find that the polycrystalline curve also has a plateau roughly corresponding to that for the single crystals, and the plateau occurs at roughly the same (corrected) stress. This is a very important result because it means that the results and implications of our studies on monocrystals can be carried over to commercial metals.

We continued to explore the problem of relating the cyclic flow stresses of single crystals and polycrystals. Since PSB behavior is single slip at least in the plateau, we wondered whether the Sachs factor could simply connect the flow stresses of the two types of material. The regular value for the Sachs factor (random polycrystalline texture) is 2·24 but the observed factor for Al-Cu containing shearable θ'' particles is 3·22. In order to check

whether the back stress acting on the PSB's due to adjoining grains was the cause of the difference, we treated the PSB as a strained ellipsoid and used Eshelby's theory to calculate the back stress. It turns out that the back stress is very low because of the narrow width of the PSB, and this could not explain the discrepancy. Another possible explanation is that the polycrystal is textured in such a manner that the Sachs factor is increased. With the kind assistance of Professor Starke, a pole figure for the alloy was measured but unfortunately the grain size was too large to give an unequivocal result. Accordingly, we used Laue back reflection X-ray technique to measure the orientation of individual grains in the cross-section. The average Schmid factor for the primary systems in these grains came to 0.47 so the usual Sachs factor applied. Clearly then, the Sachs factor does not explain the connection between mono- and polycrystalline behavior in age-hardened Al-Cu alloy. It is interesting that the observed factor, as noted above, is quite near to the Taylor's factor with a slight increment due to the back stress from adjoining grains. Accordingly we feel that incompatibility due to adjoining grains has in fact modified the dislocation structure of the PSB's in the polycrystal with respect to that in single crystals. This analysis was written up for presentation at the September 1981 Denmark conference(5), and in the last year's report, we noted that another paper would be required to document fully the results on Al-Cu polycrystals.

With the help of Drs. Horibe and J. K. Lee, the last post-doctoral students employed in this project, we have completed the polycrystalline studies. The issue was whether or not the cyclic

deformation of Al-Cu containing θ" precipitates would show a plateau in the cyclic stress-strain curve, if the grain size was small. (The polycrystalline material referred to above which did show a plateau had a large grain size, ~1 mm). We have now shown that the plateau is indeed suppressed for small grained material, just as it is in pure metal, and the promised paper interpreting the behavior has been completed(6).

We have made good progress in studying persistent slip band behavior by "Finney-type" interferometry(7); i.e., we cycled a specimen to a desired point in its life, interrupted the test and removed the specimen from the machine, microscopically recorded the slip band structure, polished the specimen, subjected it to a quarter cycle of strain to reveal the <u>current</u> state of slip behavior (as opposed to the accumulated behavior), and then studied the slip localization by interferometry. One side of the specimen was not polished so that the accumulated slip band behavior could be compared with the current behavior. We have thus proved that PSB's do not form until just before the peak and we have gathered observations on slip behavior during softening. It turns out that the interferometric observations can be done most successfully by means of replicas provided the trick is known of preventing the replicas from curling. By TEM observations, the width of the slip bands is known, and from measurements of slip offsets, the details of the strain localization can be measured. The strain is extremely large, ranging from 0.3 to 0.6. The volume fraction of PSB's is quite low throughout the plateau regime of strain, the order of a few percent, and the localization becomes more intense with cyclic softening.

The number of active bands remains constant during softening and thus the increased localized strain is obtained at the expense of the matrix. PSB's continue to operate at strains greater than those of the plateau but we observed them to form on intersecting planes. For strain amplitudes up to 0.01, the highest we studied, the volume fraction of PSB's remains low, however.

TEM observations of the Al-Cu alloy have been comp ed; the PSB structures in specimens cycled to failure have bee bserved on two orthogonal sections. The dislocation structures h been revealed and the θ" precipitates observed to survive th cycling. A ball-model experiment of θ" behavior has been completed in conjunction with another project and has been useful in assessing the kinetics of disordering(4).

Also during the program we were fortunate to obtain a mature Masters student, Alain Renard, working with a French Government Scholarship and the support of the Pechiney Co., to complete our studies of commercial aluminum alloys and polycrystalline Al-Zn-Mg. He studied the cyclic response of certain of the Frankford/Picatinny alloys (courtest J. Waldman), including 7075 in the T6 and TMT conditions, both of commercial purity and high purity with respect to constituent particles. No plateaus have been found for these alloys. The Al-Zn-Mg alloy has been studied in two conditions of ageing both to make connections with the 7075 alloy and also with the studies by Wilhelm on monocrystalline alloy(8). The low strain amplitudes used have been a particular feature of this work.

It will be further recalled that three other projects were proposed for the penultimate year of the program: a) completing our

studies of Al-Cu alloy containing θ", b) studying the response of monocrystalline Al-Cu containing θ' precipitates and more complex arrangements of precipitates and c) doing some variable amplitude tests of Al-Cu containing θ". These items have now been completed, and publications reporting the results are either complete or in the final stages of preparation. As an example of this work, consider our investigation of Al-Cu single crystals containing θ' precipitates; we focused on the behavior in cyclic deformations, and observed the following: No plateaus were observed in the cyclic stress-strain curves of this material because the non-shearable precipitates homogenized the strain and prevented strain localization. At low strain amplitudes, for constant amplitude tests, the flow stresses were higher in multislip crystals as compared to crystals in single-slip orientations during the initial cycles, but we were astonished to find that the saturation stresses were lower. We explained this behavior by the effect of secondary dislocations which were multiplied in the multi-slip specimens early in the tests and which reduced the energies of the primary-dominated dislocation arrays after saturation was attained. At high strain amplitudes, TEM observations showed increasing populations of secondary dislocations even in the single slip crystals, and these evened out, but did not eliminate, differences from the behavior of multi-slip crystals.

At low strain amplitudes, the dislocation structures consisted of dipolar walls, strongly dominated by primary dislocations, and located at the broad faces of the θ' plates. These structures, in their uniform and widespread nature, produced behavior typical

of that of loop patches in homophase crystals. However, they were also similar to the dipolar walls of PSB's and the dislocation behavior was concluded to be rather similar to that of PSB's, the channels between the θ' precipitates playing the same role as that of channels in PSB's.

At high strain amplitudes, where the the density of secondary dislocations increases, the θ' plates provided a skeleton on which a "maze" structure of dipolar walls (containing complex arrays with mixed Burgers vectors) was framed. Many of the features of maze structure in homophase metal appeared quite similar to those observed in the Al-Cu alloy of interest here. The dislocation arrays which formed at the broad faces of the θ' plates served to protect them from dislocation cutting, which was investigated by dark field TEM technique. Since the θ' crystal is ordered, its cutting by a matrix dislocation leaves an APB which can be observed. We concluded that dislocation cutting occurred hardly at all in crystals oriented for single slip, and only early in life for multi-slip crystals.

Many additional results were found for hysteresis loop shapes, for a variety of program loading tests, and from tests containing rests. The details are now being written up, but are too lengthy to be described here.

In parallel with the work on Al-Cu, we studied MAR-M-200 nickel-based superalloy single crystals. We initially attempted a "Finney-type" interferometric study but the material was too strong for this to be feasible. Instead we completed mechanical studies under plastic strain control (the behavior is quite similar to that of Al-

Cu) and replication studies (again with great similarity to that of Al-Cu but with nothing like the detail). We were disappointed to run out of material when the results were getting interesting. In this connection Wilhelm's mechanical study of Al-Zn-Mg alloy single crystals containing precipitates(8) should be noted. Although he did not make our broader connections to fracture mechanisms, slip band behavior, and to polycrystals, his results were sufficiently similar to ours on Al-Cu to encourage us in the general significance of our conclusions. Therefore in the last year of our project we emphasized a study of Ni-based alloy single crystals which we believed would confirm the generalities we have claimed from our work on Al-Cu. The details are as follows:

As proposed last year, we were greatly excited by a paper by Jablonski and Sargent(9) who showed that significant anisotropic fatigue hardening occurred in a nickel-based superalloy at 760°C when cycled in strain control at a constant strain amplitude of 1.0%. These workers noted that the maximum stress amplitude increased 270 MPa between cycles 500 and 10^4 while the mean stress increased 550 MPa during the same interval. They attributed this astonishing phenomenon to dislocation image stresses which aided dislocation motion in compression.

We were skeptical of this interpretation, and brought the results to the attention of our colleagues, Ezz and Pope, who provided a thorough theoretical model based on yield stress anisotropy observed in Ni (Al, Nb) single crystals to explain the observations(10). This yield stress anisotropy, based on the relative Shockley partial dislocation spacing in screw super dislocations

was first introduced by Lall et al(11), again in Ni (Al, Nb) single crystals. This model predicts that compressive glide would be enhanced for a stress axis of [100] while a stress axis [111] should reverse this asymmetry. The single crystal approach to a precipitation hardened system was made by keeping in mind the large γ' volume fraction (~60%) in the Jablonski-Sargent alloy.

While we were troubled by the Jablonski-Sargent results, because much work had been done in similar systems, especially MAR-M-200, without any hint of anisotropic fatigue hardening being detected, we were nevertheless persuaded that the Ezz and Pope experiments, in conjunction with modelling being done in collaboration with Vitek, were compelling enough to make the Jablonski-Sargent results reasonable.

Accordingly we tested Ni (Al, Nb) single crystals of γ' and Udimet 700, both to check anisotropic fatigue hardening, to explore the possibility of shape changes due to anisotropic deformation, and to find out how PSB's behave in these materials. Using Udimet 700, we were able to reproduce at room temperature the results of Jablonski and Sargent; this troubled us greatly because the texture of the alloy was not sufficiently marked to give rise to anisotropic fatigue hardening. By further experimentation we discovered that the anisotropic behavior was caused by slippage of the extensometer. Since we were using our customary techniques which had previously given us no problems, we explained the behavior by the great hardness of the alloy which prevented the penetration of the V-chisel gage-definers. In the softer alloys studied previously, penetration of the extensometer had prevented slippage.

Anton has also published a comment on the Jablonski-Sargent results with conclusions similar to our own(12). This comment was especially compelling because Anton used the same equipment as used by Jablonski and Sargent. Nevertheless, Ezz and Pope have continued their experiments on Ni (Al, Nb) single crystals and have demonstrated decisively the existence of anisotropic hardening.

We have begun gathering data on strain localization in PSB's, using Ni (Al, Nb) single crystals but the investigation is still in a relatively early stage. Development of this program in suitably detailed fashion is proposed for future work.

Publications Resulting from the Above Described Program of Research:

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2) J.K. Lee, S.P. Bhat, R. de la Veaux and C. Laird, "Mechanisms of Cyclic Softening in Precipitation Hardened Alloys--A Ball Model Approach and Tests at 78K", Int. J. of Frac., 17, 1981, 121.

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J. K. Lee - graduate student, obtained Ph.D. degree (post-doctoral student for a short period).

A. Renard - graduate student, obtained MS degree.

S. Horibe - post-doctoral student.

Bonda Rao - graduate student - course of study not yet finished.

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